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Effect of Composition on Grain Growth in Aluminum-magnesium Solid Solutions

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(Philadelphia Meeting, October 1948)

As reported in a previous publication,[1] isothermal grain growth in high purity aluminum and in an aluminum alloy with 2 pct magnesium can be adequately described by means of the empirical relation:

$$D = \frac{D_r}{A^n} (t_g + A)^n \qquad [1]$$

or in some cases, by the simpler relation,

$$D = \frac{D_r}{R^n} t^n \qquad [2]$$

Here, D is the average grain size after an annealing period t. The annealing period includes the time for complete recrystallization R, and the time for grain growth t_g. D_r is the grain size as recrystallized, n is a parameter depending on the temperature and on the material. The effect of 2 pct magnesium in solid solution was to decrease D_r and to increase R, A and n at a given temperature.

The present work was undertaken in order to investigate the above listed effects of magnesium in solid solution, as functions of the magnesium content. The work also includes the study of certain anomalous effects observed in the recrystallization and grain growth of the 2 pct magnesium alloy at 350°C.

Experimental Procedure

The method of producing the ingots used in this work was described in detail[1] and need not be reproduced here. The materials used were high purity aluminum, furnished by the Aluminum Co. of America, of lot analysis shown in Table 1 and magnesium from a high purity distilled stock of magnesium crystals.

Table 1—*Lot Analysis of High Purity Aluminum*

	Per Cent
Si	0.002
Fe	0.002
Cu	0.002
Mg	0.003
Mn	0.001

Ingots were prepared for a series of binary aluminum-magnesium alloys of increasing alloy content. The numbers used to designate these ingots and their magnesium contents by chemical analysis are given in Table 2.

Table 2—*Ingot Numbers and Magnesium Contents*

Ingot Number	Pct of Magnesium
21	2.05
22	1.80
24	0.12
26	0.025

The analyses showed no difference in composition between the top and the bottom of the ingots. Spectrographic analysis of the ingots for elements other than magnesium showed, within the limits of accuracy of the method, no increase in impurities over the amounts given in the lot analysis for the high purity aluminum in Table 1. Careful microscopic study of ingot 21 in the as-cast condition, at magnifications up to 1500×, revealed a cer-

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[1] References are at the end of the paper.

tain amount of coring, but no trace of the β(Al,Mg) phase.

The coarse grained ingots were broken down into uniformly fine grained strips by a series of alternate rolling and annealing operations which led to successively finer grain sizes. It was found that a series of 33 pct reductions by rolling resulted in the most uniform grain size. The rolling and annealing schedules for ingots 21 and 22 have been given.[1] The schedules for ingots 24 and 26 are given in Table 3. In all cases the final reduction by rolling was also 33 pct.

TABLE 3—*Rolling and Annealing Schedules for Ingots 24 and 26*

All ingots were 1½ in. in diam as cast

Rolled to in.	Annealing Time, min.	Annealing Temperature, °C
For Ingot No. 24		
1.035	60	600
0.800	30	400
0.550	30	400
0.360	30	400
0.240	30	400
0.160	30	350
0.095	30	350
0.063	Specimens cut from strip as rolled	
For Ingot No. 26		
1.035	60	600
0.800	30	400
0.550	30	400
0.360	30	400
0.240	30	400
0.160	30	350
0.100	30	350
0.067	Specimens cut from strip as rolled	

Specimens were cut from the strips and annealed in electrically heated and controlled salt baths at temperatures of 350 to 600°C in 50°C steps for periods of time of 20 sec, 1, 5, 25, 125, 635, 3125 and 15625 min. After annealing, the specimens were quenched immediately in water at room temperature. They were then deep etched to reveal their grain structure with the etchant given in Ref. 1.

The grain size determinations were made in the same manner as that described[1] and again the "extremely probable range" for the mean grain diameter was determined and plotted on the graphs for specimens with mean grain diameters less than about 0.7 mm. For the larger grained specimens only one tracing (instead of ten) was made, The area traced, however, was large enough to include most of the grains in the specimen. No range could be determined in these cases.

It was noted that the grain size of the specimens changed with distance from the rolled surface, reaching a constant value for the 0.020 in. specimens at about four or five thousandths of an inch below the surface, but increasing to the center of the specimens of 0.063 in. in thickness and above. The thinner specimens, therefore, were etched to one-half their thickness before making the grain size determinations and the thicker specimens were milled, ground, and etched on one side so that the determinations could be made at the center of the specimens.

The periods of time required for complete recrystallization were determined for the three alloys at temperatures of 350 and 400°C and for specimens made from the alloy of highest magnesium content, ingot 21, also at 375°C.

EXPERIMENTAL RESULTS

The isothermal grain growth data for the alloy Al + 2.05 pct Mg, with specimens of 0.020 in. in thickness, are presented in Fig 1, in which the logarithm of the mean grain diameter D in mm is plotted against the logarithm of the total annealing time t in minutes. These data were given previously[1] except for the lines at 390, 375 and 350°C. The outstanding features of the graphs shown in this figure were discussed previously.[1] They are: (1) the $D = kt^n$ type of relationship between the mean grain diameter D and the annealing time t, (2) the specimen thickness effect causing a stoppage of grain growth at values of mean grain diameter just slightly larger than the specimen thickness, and (3) the increase of the exponent n

of the above equation with the annealing temperature. In Fig 1 the dotted lines show deviations from the straight line relationship at the longer time periods for expected. A check specimen was annealed after remaining three months longer at room temperature in the as-rolled condition and showed a grain diameter much

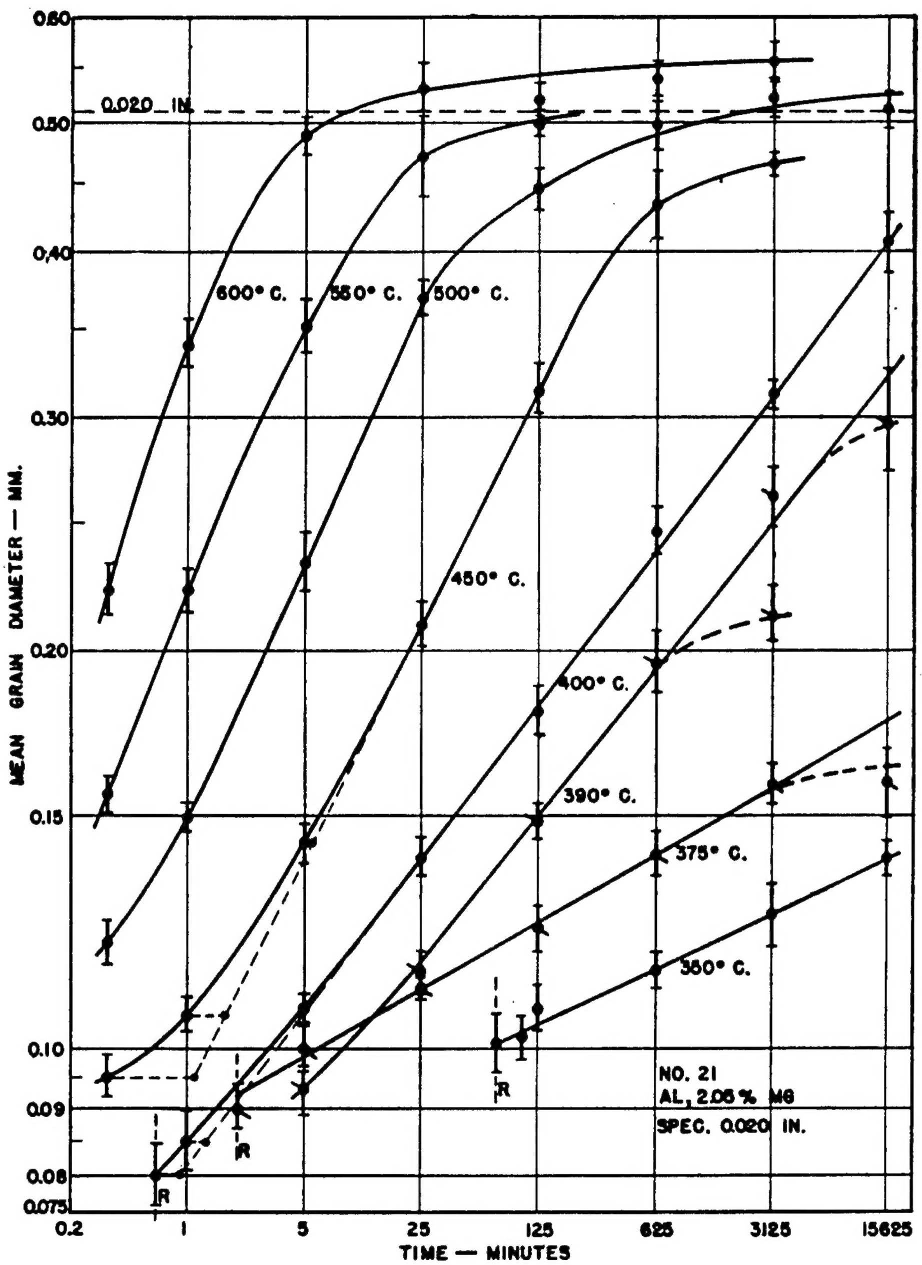


FIG 1—AVERAGE GRAIN DIAMETER (LOG. PLOT) IN Al + 2 PCT Mg ALLOY VS. ANNEALING TIME (LOG. PLOT) AT 350, 375, 390, 400, 450, 500, 550, AND 600°C. 33 PCT REDUCTION BY ROLLING. SPECIMEN THICKNESS 0.020 IN.

temperatures of 390 and 375°C. There are two points plotted at 3125 min. for the 390°C curve. The specimen annealed together with the shorter period specimens at 390°C showed a grain size smaller than closer to the straight line connecting the points at the shorter time periods. The points at 15625 min. at temperatures of 390 and 375°C also showed similar deviations from the straight lines, but checks,

similar to the above, were not made at these periods.

The variation of n (the slope of the lines in the log-log plot) with temperature is lower lines are replots of the data of Fig 1 for the 0.020 in. specimens. In Fig 1 and 2 the points marked R indicate the condition of just complete recrystallization.

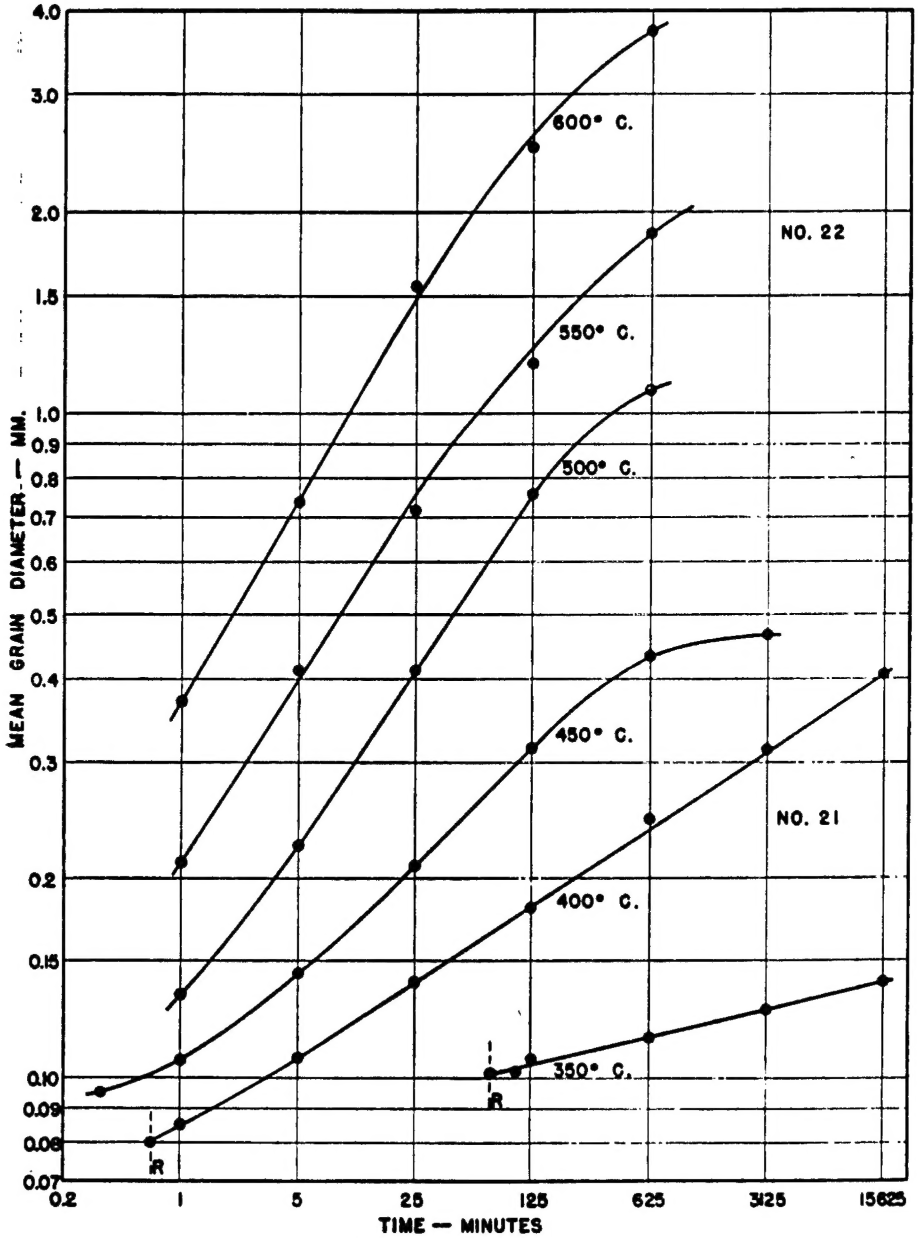


FIG 2—AVERAGE GRAIN DIAMETER (LOG. PLOT) IN Al + 2 PCT Mg ALLOY VS. ANNEALING TIME (LOG. PLOT) AT 350, 400, 450, 500, 550, AND 600°C. 33 PCT REDUCTION BY ROLLING. SPECIMEN THICKNESS 0.020 TO 0.160 IN.

shown more clearly in Fig 2 where specimens made from ingot 22, Al + 1.8 pct Mg, were used at a thickness of 0.160 in. for the 600 and 550°C lines and at a thickness of 0.100 in. for the 500°C line. The three

The data for the alloy, Al + 0.12 pct Mg, obtained with specimens 0.063 in. in thickness, are given in Fig 3. Here again the conformity of the data to the $D = kt^n$ type of relationship is apparent, as is also

the presence of the specimen thickness effect in the lines for the temperatures of 450°C and above. A definite increase of the slope of the lines with the temperature shape to those for the alloy of intermediate magnesium content, but again the decrease in percentage of alloying element has resulted in a decrease in slope of the curves

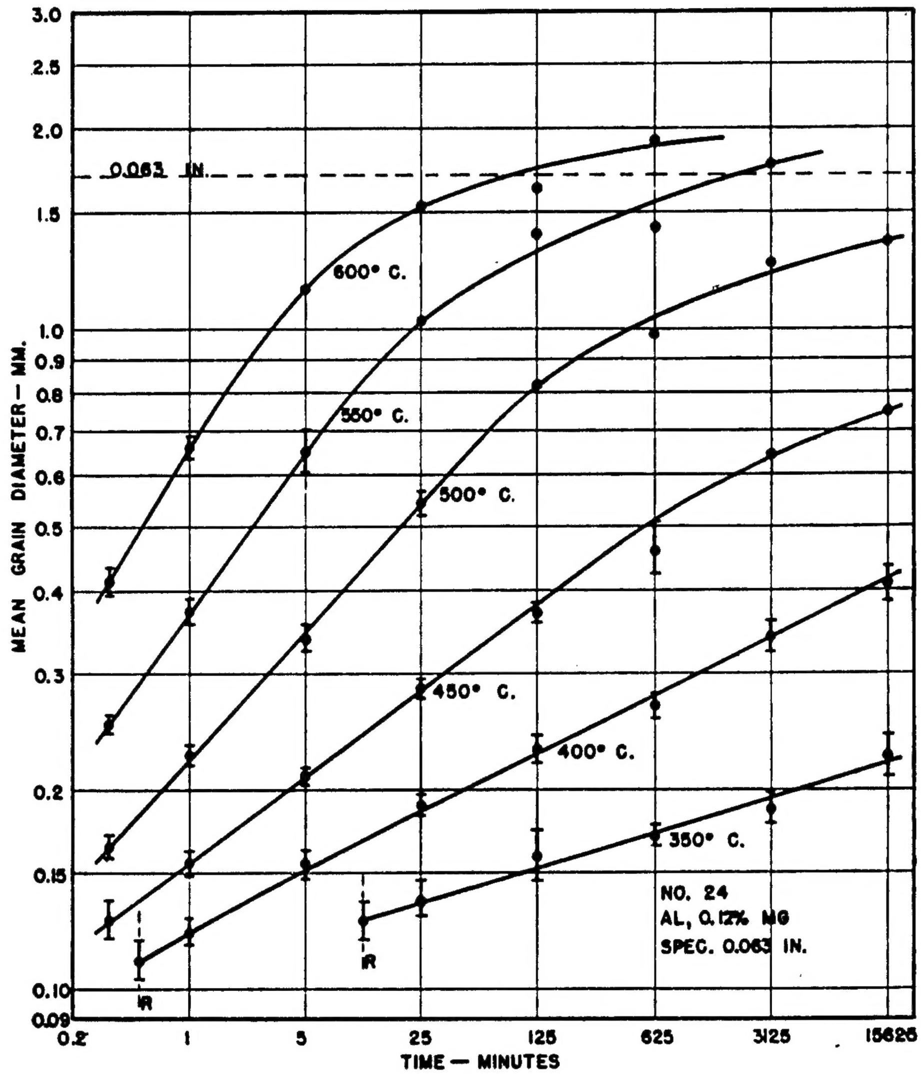


FIG 3—AVERAGE GRAIN DIAMETER (LOG. PLOT) IN Al + 0.12 PCT Mg ALLOY VS. ANNEALING TIME (LOG. PLOT) AT 350, 400, 450, 500, 550 AND 600°C. 33 PCT REDUCTION BY ROLLING. SPECIMEN THICKNESS 0.063 IN.

may be noted. It should also be observed, in comparing Fig 2 and 3, that the lines for the alloy of lower magnesium content show a lesser slope than the corresponding lines for the high magnesium alloy, except for the line for 350°C.

In Fig 4 are given the data for the Al + 0.025 pct Mg alloy (from ingot 26), obtained with specimens 0.067 in. in thickness. These lines are seen to be similar in for corresponding temperatures. Deviations from the straight line relationship in the specimens at the longest periods for temperatures of 450 and 400°C are shown by dotted curves in Fig 4. The point at 3125 min. at 450°C was re-run and the same value obtained with another specimen but the two points at the longest periods were not checked. The significance of these deviations is not clear.

The n values for the three alloys, as determined from the slopes of the lines in Fig 1, 2, 3, and 4, are given in Table 4 and are plotted in Fig 5 to a logarithmic points below 500°C satisfy a straight line relationship with the line almost parallel to that for high purity aluminum. Above 500°C the curve bends toward the line for

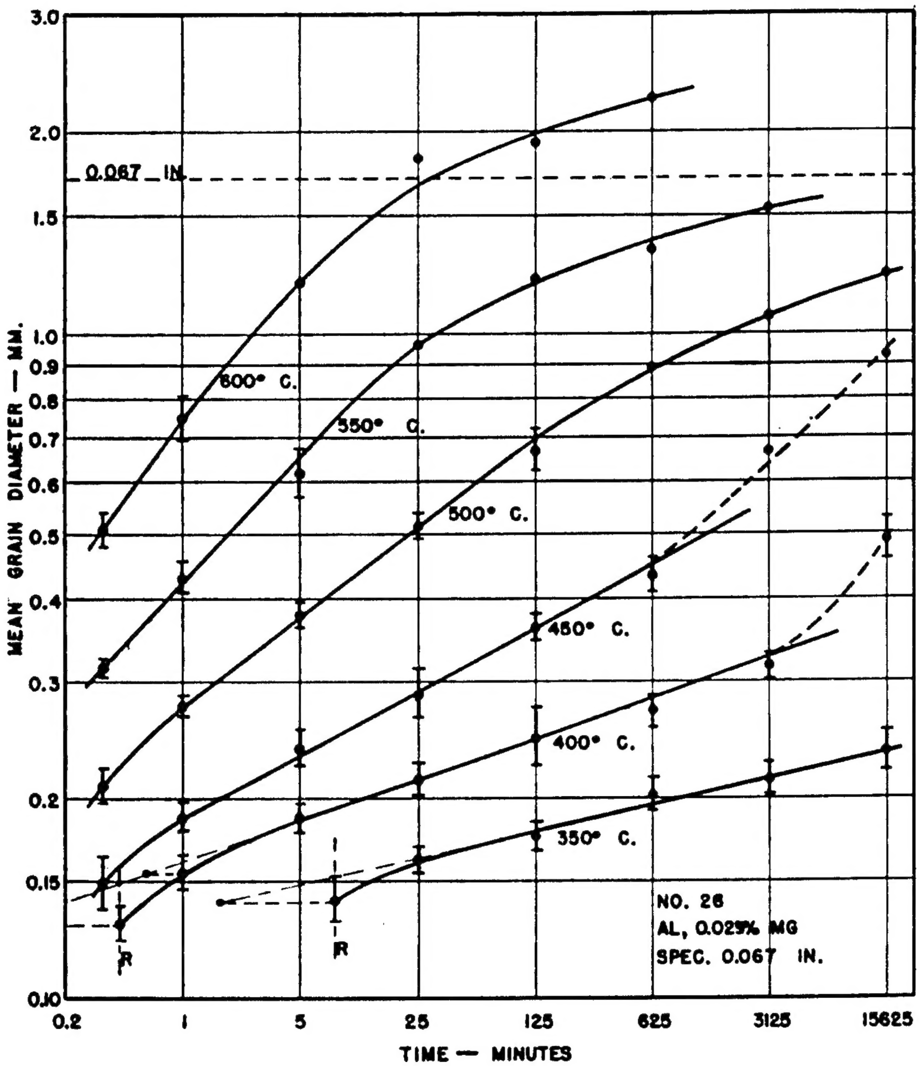


FIG 4—AVERAGE GRAIN DIAMETER (LOG. PLOT) IN Al + 0.025 PCT Mg ALLOY VS. ANNEALING TIME (LOG. PLOT) AT 350, 400, 450, 500, 550 AND 600°C. 33 PCT REDUCTION BY ROLLING. SPECIMEN THICKNESS 0.067 IN.

scale versus the reciprocal absolute temperature marked in degrees Centigrade. The data for high purity aluminum[1] are also shown in Fig 5. Curve B for the Al + 0.025 pct Mg alloy is very close to, and only slightly higher than, curve A for high purity aluminum. A decided increase in the slope value for each temperature is seen to result from an increase in alloy content to 0.12 pct Mg (Curve C). The high purity aluminum. This behavior is even more pronounced with the alloy of highest magnesium content (curve D). The tendency for the Al-Mg solid solution alloys to approach the same n value as pure aluminum, approximately 0.45, near the melting point of aluminum, which was discussed previously,[1] is thus confirmed.

The n values for the alloy highest in magnesium, line D in Fig 5, show consid-

erable increase over those for the alloy of intermediate magnesium content at most temperatures. However, the sudden drop of curve D at low temperatures deserves considerable attention. Between the temperatures of 375 and 390°C there is a sharp decrease in the n value for the highest magnesium alloy, so that the slope values at 350 and 375°C are distinctly lower than expected from extrapolation of the line connecting the points between 390 and 500°C. In fact, the n values at 350 and 375°C are only slightly higher than the corresponding n values for high purity aluminum. This curious behavior is also apparent in Fig 1 where the great decrease in slope between the curves at 390 and 375°C is striking. Discussion of this effect is given further below.

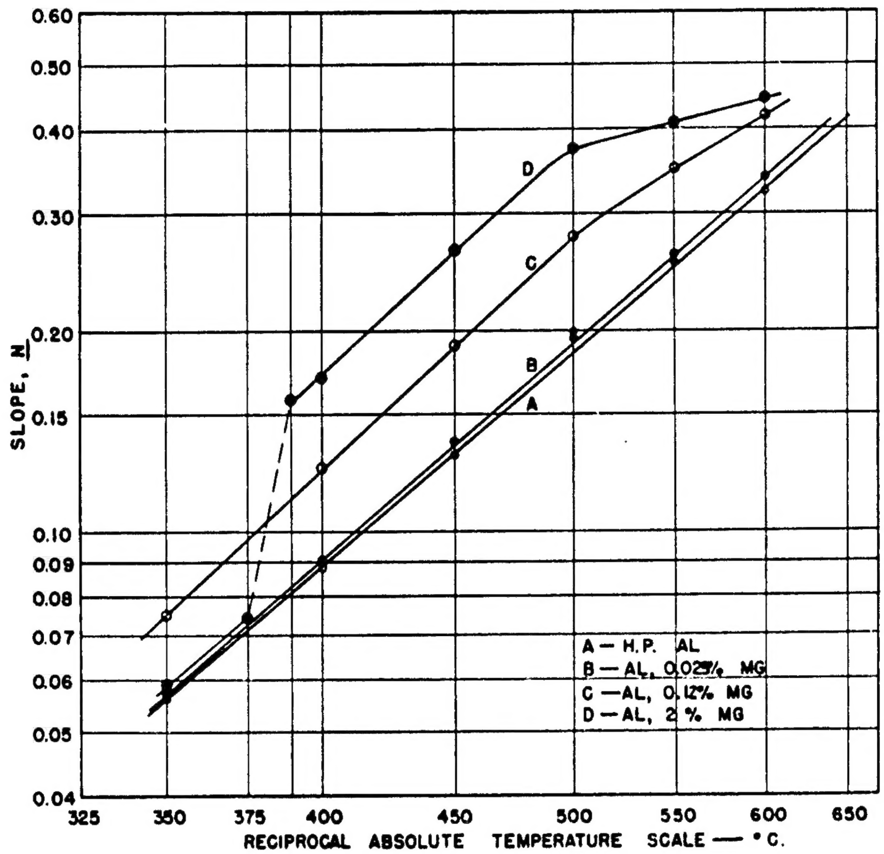


FIG 5—EXPONENT n (LOG. PLOT) VS. TEMPERATURE (RECIPROCAL ABSOLUTE TEMPERATURE PLOT) FOR HIGH PURITY ALUMINUM AND 3 ALUMINUM-MAGNESIUM ALLOYS.

TABLE 4—*Values of n and A.*

°C	0.025 pct Mg.		0.12 pct Mg.		2 pct Mg.	
	n	A min.	n	A min.	n	A min.
350	0.057	1.7	0.075	11	0.059	66
375					0.074	
390					0.158	3.3*
400	0.090	0.08	0.125	0.5	0.170	0.8
450	0.136		0.190		0.265	0.16*
500	0.194		0.277		0.376	
550	0.260		0.350		0.410	
600	0.340		0.420		0.445	

* Determined from D_r values obtained by linear extrapolation according to Fig 14.

The relative effect of the different magnesium additions is shown in Fig 6 where the values for the slope n are plotted to a linear scale versus the magnesium content to a logarithmic scale. The data for high purity aluminum[1] are also plotted on this graph; the abscissa corresponds to a magnesium content of 0.003 pct, the amount of

magnesium given in the lot analysis for the high purity aluminum used. The points for the alloy of highest magnesium content are plotted at 1.8 pct for the three highest temperatures, as they were determined using ingot 22, while the points at the three lower temperatures, determined using ingot 21, are plotted at 2.05 pct Mg. The graph shows that an increase of the magnesium content from 0.003 to 0.025 pct causes very little change in the exponent *n*. At 400, 450 and 500°C the *n* value, between 0.025 and 2 pct magnesium, increases approximately linearly with the logarithm of the magnesium content. It should be noted that the deviation from linearity at 0.12 pct magnesium is not very large, but it is systematic, that is, toward high *n* values at all three temperatures. The unexpectedly low position of the point for 350°C for the alloy of highest magnesium content is again apparent in this plot. At 550 and 600°C great deviations from the linearity occur. These deviations result from the tendency of the high magnesium alloys to approach the same *n* value as high purity aluminum near the melting point of aluminum, as shown by curve *D* in Fig 5.

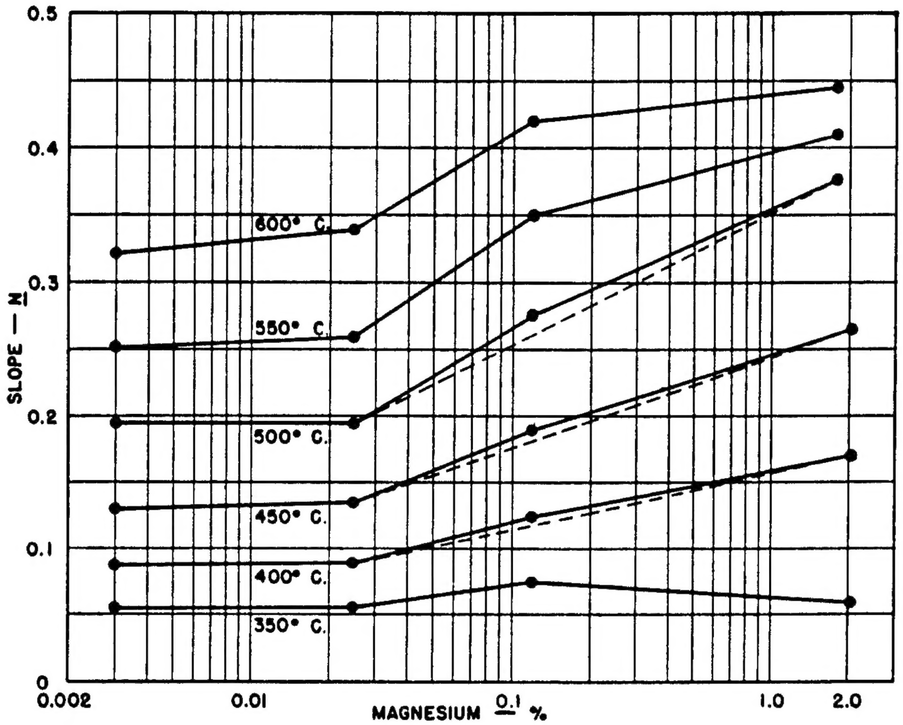


FIG 6—EXPONENT *n* VS. MAGNESIUM CONTENT (LOG. PLOT) FOR VARIOUS ANNEALING TEMPERATURES.

In addition to the *n* values, Table 4 also gives the *A* values determined, for the lowest temperatures, from the intersection of the extrapolated straight line portion of the logarithmic grain growth lines in Fig 1, 3, and 4, with the horizontals corresponding to the grain size as recrystallized at each temperature. These *A* values for the three alloys are plotted in Fig 7 to a logarithmic scale vs. the reciprocal absolute temperature. The points are connected by straight lines which have the same slope for all three alloys. The 400°C point for the Al + 2 pct Mg alloy is unexplainably low. The 350°C point for the same alloy is high, in line with the abnormally long period of recrystallization discussed below. Fig 8 shows *A* at 350 and 400°C as a function of the magnesium content. The 2.05 pct Mg points on the solid curves correspond to the straight line in Fig 7; the

actually observed abnormally high A value at 350°C is used for the dotted line.

The recrystallization data obtained in this work are presented in Table 5. The effect of magnesium content on the time required for complete recrystallization is shown graphically in Fig 9 and 10 in which the time necessary for complete recrystallization is plotted linearly, versus the magnesium content, plotted to a logarithmic scale. The data for high purity aluminum are again taken from Ref. No. 1 and are plotted at a magnesium content of 0.003 pct for the reason given above. At 400°C the time required to attain complete recrystallization is seen to vary in a logarithmic manner with the magnesium content. At 350°C the straight line relationship on the semi-log plot is again satisfied for all but the alloy of highest magnesium content. The exceptionally long time for complete recrystallization for this alloy at 350°C should be noted in connection with the unexpectedly low value for the slope of the logarithmic grain growth line at this temperature as shown in Fig 6.

The mean grain diameter of each alloy in the just completely recrystallized condition is given in Fig 11 to a linear scale vs. the magnesium content to a logarithmic scale for temperatures of 350 and 400°C. A definite decrease in recrystallized grain size with increasing magnesium content is apparent. Another interesting feature noticeable in this plot is the fact that for all alloys the grain size, as recrystallized, is smaller for the higher temperature of recrystallization.

Discussion of Results

The effect of the magnesium content on the grain size of the Al-Mg solid solution alloys is a complex one. Magnesium in

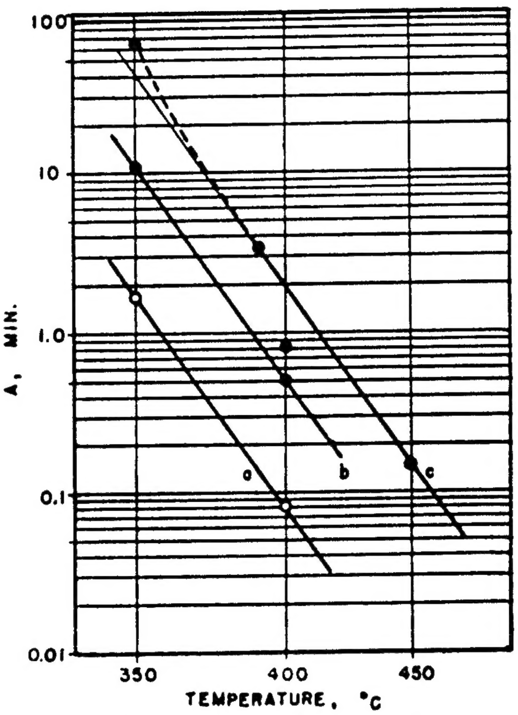


Fig 7—Parameter A (log. plot) vs. temperature (reciprocal absolute temperature plot) for 3 aluminum-magnesium alloys.

solid solution *decreases* the grain size as recrystallized (D_r) and *increases* the exponent n in the isothermal grain growth formula: $D = \frac{D_r}{A^n}(t_g + A)^n$. Both effects are shown by the present data to be very small up to a magnesium content of 0.025 pct. Between 0.025 and 2 pct magnesium both effects vary approximately linearly with the logarithm of the magnesium con-

Table 5—*Time Required for Complete Recrystallization and Recrystallized Grain Diameter*

Temperature of Recrystallization °C	H.P. Al*	Al 0.025 pct Mg	Al 0.12 pct Mg	Al 2.05 pct Mg
350	Time 4.7 ± 0.3 min. Gr. Diam. 0.141 mm	8 ± 1 min. 0.140 mm	11 ± 1 min. 0.127 mm	66 ± 4 min. 0.101 mm
375	Time Gr. Dia.			3.5 ± 0.5 min. 0.0190 mm
400	Time 20 ± 2 sec. Gr. Diam. 0.130 mm	25 ± 3 sec. 0.129 mm	30 ± 3 sec. 0.110 mm	40 ± 5 sec. 0.080 mm

* From Ref. No. 1.

tent (Fig 6 and 11), at least in certain temperature ranges. The time for complete recrystallization R increases approximately linearly with the logarithm of the magnesium content at 400°C and partly also at 350°C. The anomalous behavior of the 2 pct magnesium alloy at 350°C merits detailed discussion.

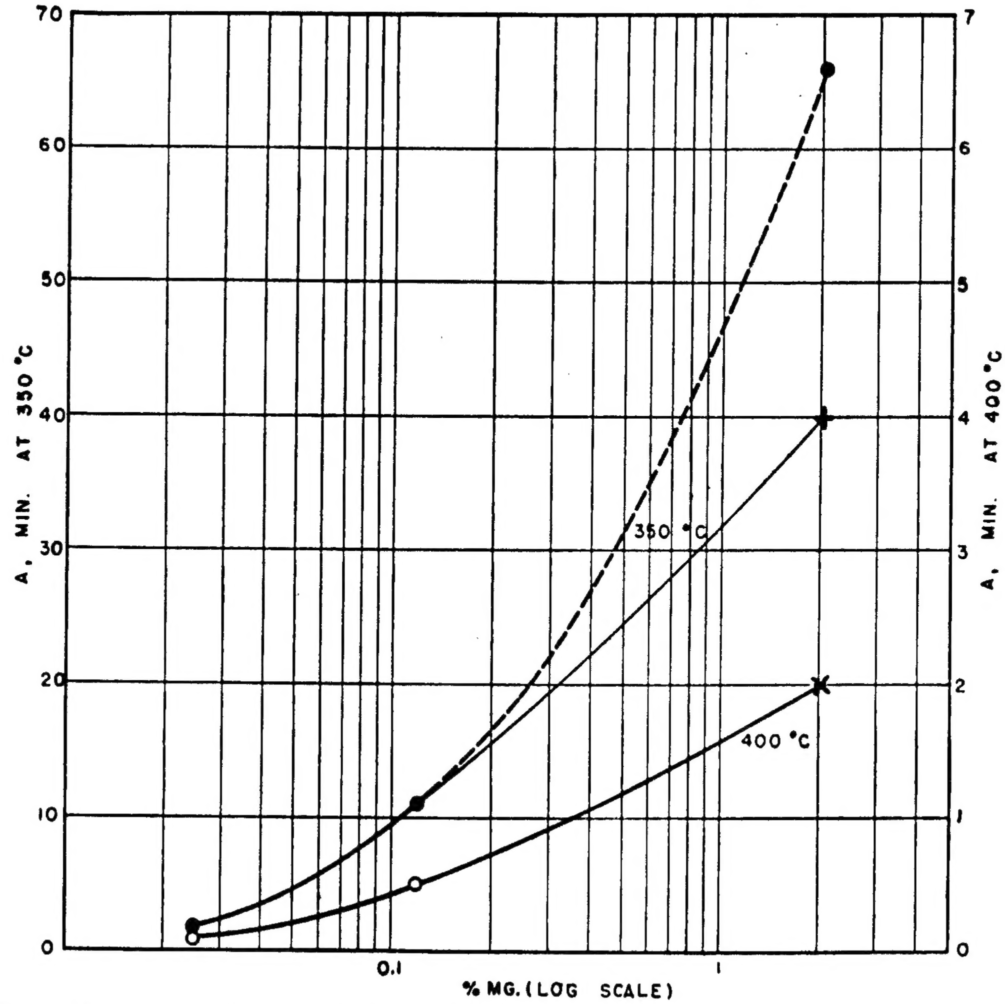
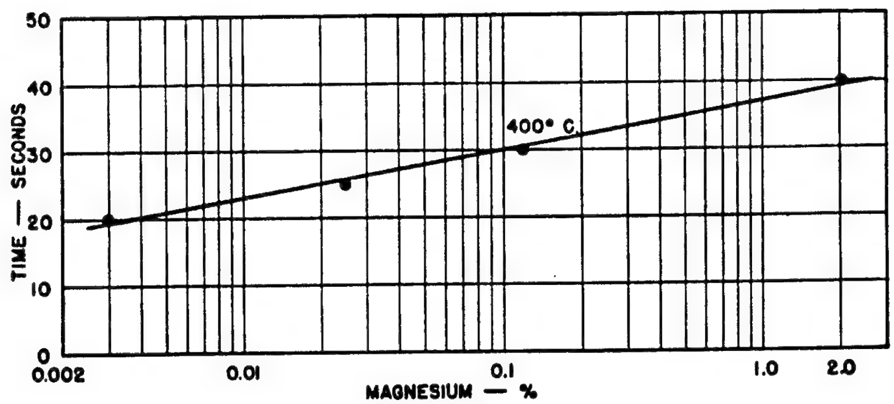


FIG 8—PARAMETER A VS. MAGNESIUM CONTENT (LOG. PLOT) AT 350 AND 400°C. POINTS MARKED ● WERE OBTAINED FROM EXPERIMENTAL RESULTS. POINTS MARKED + AND X WERE LINEARLY EXTRAPOLATED FROM FIG 7.

The limit of solid solubility of magnesium in aluminum, according to Fink and Freche,[2] can be extrapolated to about 0.15 pct at 20°C and it is 9 pct at 350°C. Because of difficulty in obtaining equilibrium, however, an alloy of Al + 2 pct Mg has been considered, in practice, to be a solid solution alloy at room temperature. Because of the irregular behavior of this alloy at 375 and 350°C in regard to both grain growth and the time required for complete recrystallization, the possibility of the presence of a second phase was considered. Examination of X ray diffraction patterns obtained with the Al + 2 pct Mg alloy gave no clue. However, careful microscopic study of specimens polished by means of the Buehler-Waisman electrolytic polisher, using a nitric acid-methyl alcohol electrolyte, gave considerable support to the assumption that precipitation does occur in this alloy. Fig 12 is a micrograph taken at 1500X with oblique illumination of a specimen made from ingot 21 by alternate

cold rolling and annealing at 350°C according to Table 2 of Ref. 1, and finally annealed at 350°C for 2960 min. Although the shape of the small dots in Fig 12 was not revealed even at the highest resolution obtainable with visible light, it was possible to ascertain that they are projecting above the polished surface. Thus they appear to be particles of a second phase. The number of such particles in an area of 0.15 mm² of the polished specimen surface was 424. Fig 13 shows a micrograph taken under similar conditions, of another specimen of the same alloy, cold rolled in the same manner, but finally annealed at 600°C for 195 min. In this specimen the number of particles present in the same area as above was only 13. Specimens of the Al + 2 pct Mg alloy, prepared by the same procedure, but finally annealed at 400°C for 40 sec, 25 min., and 15625 min. were examined microscopically at 1500× and the number of particles in an area of 0.15 mm² were found to be 302, 249, and 66 respectively. This would indicate the gradual re-solution of the precipitated



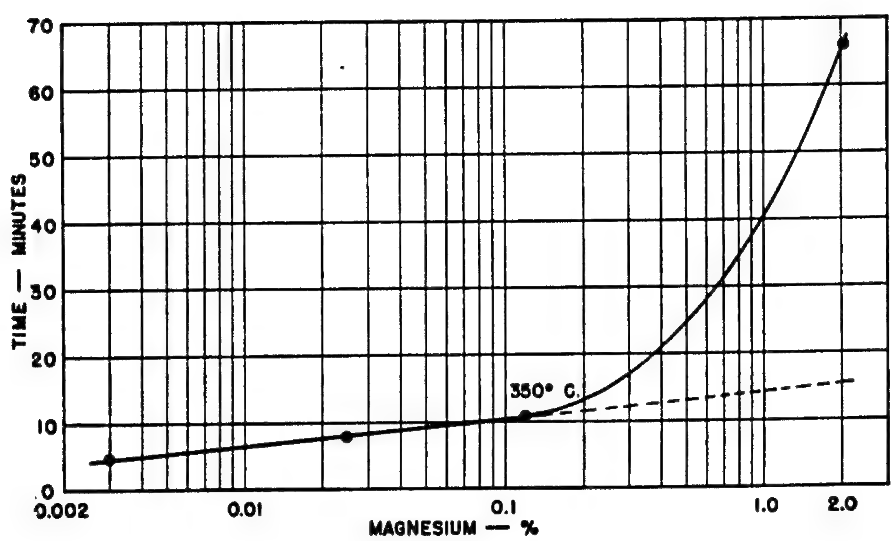


FIG 9 (top)—TIME FOR COMPLETE RECRYSTALLIZATION, *R*, VS. MAGNESIUM CONTENT (LOG. PLOT) AT 400°C.

FIG 10 (bottom)—TIME FOR COMPLETE RECRYSTALLIZATION, *R*, VS. MAGNESIUM CONTENT (LOG. PLOT) AT 350°C.

phase at 400°C. No definite conclusion as to the identity of the precipitated phase can be offered at the present time.

Fig 14 gives the logarithm of the recrystallization time as a function of the reciprocal absolute temperature for the three Al-Mg alloys and for high purity aluminum. Aside from the Al + 2.05 pct Mg alloy, which exhibits abnormally long periods of recrystallization at 350 and 375°C, "heat of activation" values Q_R may be estimated for the temperature dependence of the time for complete recrystallization R in the materials investigated. The following values were obtained from the slope of the lines in Fig 14:

44 K cal./g atom for high purity aluminum

49 K cal./g atom for Al + 0.025 pct Mg

51.5 K cal./g atom for Al + 0.12 pct Mg

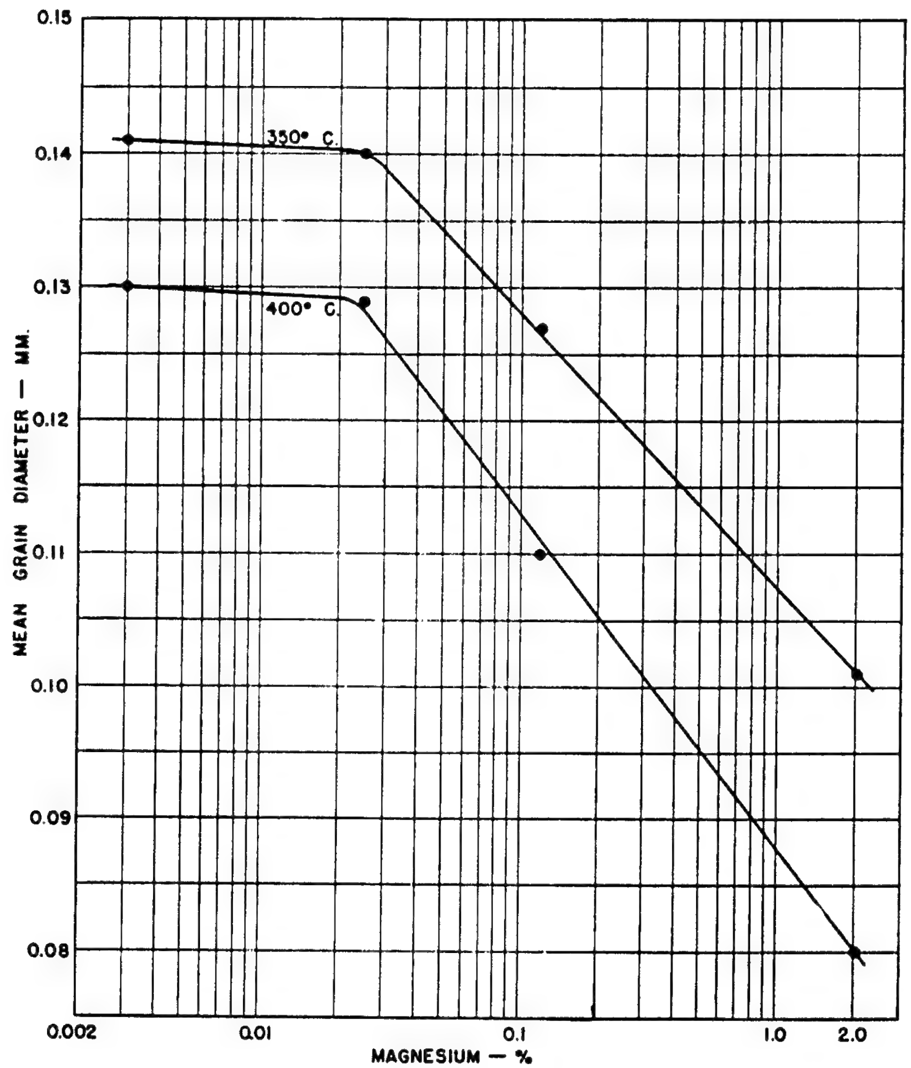


FIG 11—AVERAGE GRAIN DIAMETER AS RECRYSTALLIZED, D_r, VS. MAGNESIUM CONTENT (LOG. PLOT) AT 350 AND 400°C.

The temperature dependence of parameter A in Eq 1, determines the displacement of the starting point at D_r of the logarithmic grain growth lines with varying temperature.* Since for the alloys investigated these lines are not parallel, the displacement at D_r is different from that obtained

* For details of the interpretation of parameter A see Ref. 6.

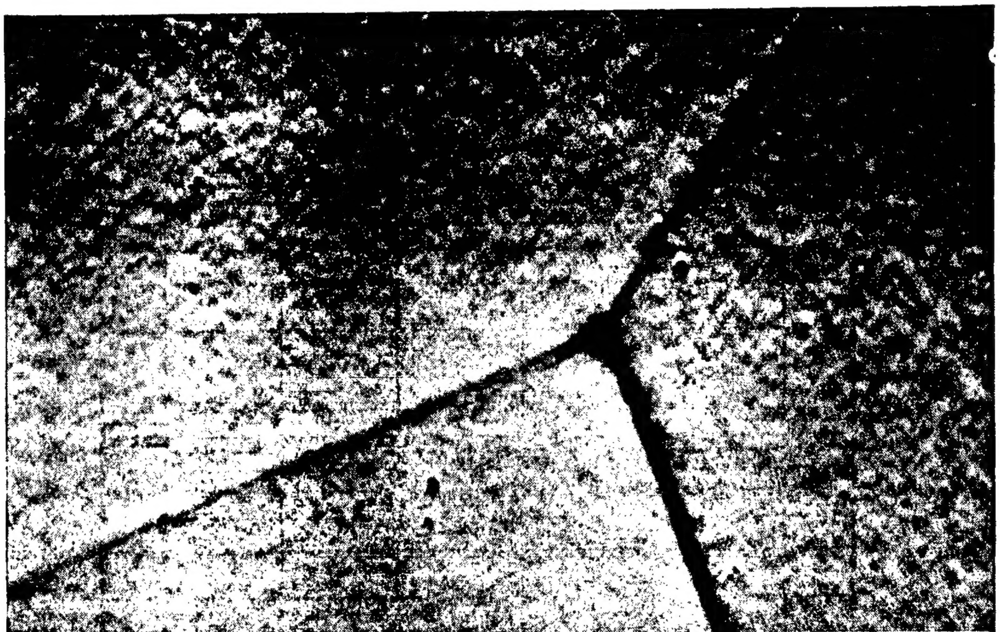


FIG 12 (top)—SECOND PHASE IN ALUMINUM + 2 PCT MAGNESIUM ALLOY ANNEALED AT 350°C FOR 2960 MIN. ELECTROLYTICALLY POLISHED, UNETCHED. MAGNIFICATION × 1500, OBLIQUE ILLUMINATION.

FIG 13 (bottom)—ABSENCE OF SECOND PHASE IN ALUMINUM + 2 PCT MAGNESIUM ALLOY ANNEALED AT 600°C FOR 195 MIN. ELECTROLYTICALLY POLISHED, UNETCHED. MAGNIFICATION × 1500, OBLIQUE ILLUMIMINION.

at larger grain sizes. As previously stated[5] for high purity aluminum, this condition precludes the determination of a definite heat of activation value Q from grain growth data, since the value which might be obtained would depend on the grain size. However, the temperature dependence of parameter A, according to Fig 7, can be used to calculate an approximate value of $Q_A = 55$ K cal/g atom, at least for the narrow temperature range of 350 to 400°C. This value is independent of the magnesium content. Comparison of Q_A with Q_R as given above, shows that they are almost equal, the difference being in the order of what may be considered their probable range of error. Although the actual values of the grain growth parameter A and of the time of recrystallization R are, in some cases, quite different, it is perhaps of significance that their temperature dependence is so similar.

Several years ago, in a paper by Eastwood, Bousu, and Eddy[3] the conclusion was reached that the grain size of alpha brass, as recrystallized, was essentially independent of the temperature of recrystallization. More recently, the work of Eastwood, James, and Bell[4] led to a similar conclusion for pure aluminum. In the discussion of the latter paper it was brought out that some decrease of the as-recrystallized grain size with increasing temperature might be expected, particularly at low degrees of deformation, and that Eastwood, James and Bell's own data offered some indication of such an effect. The present data, as given in Table 5 and in Fig 11

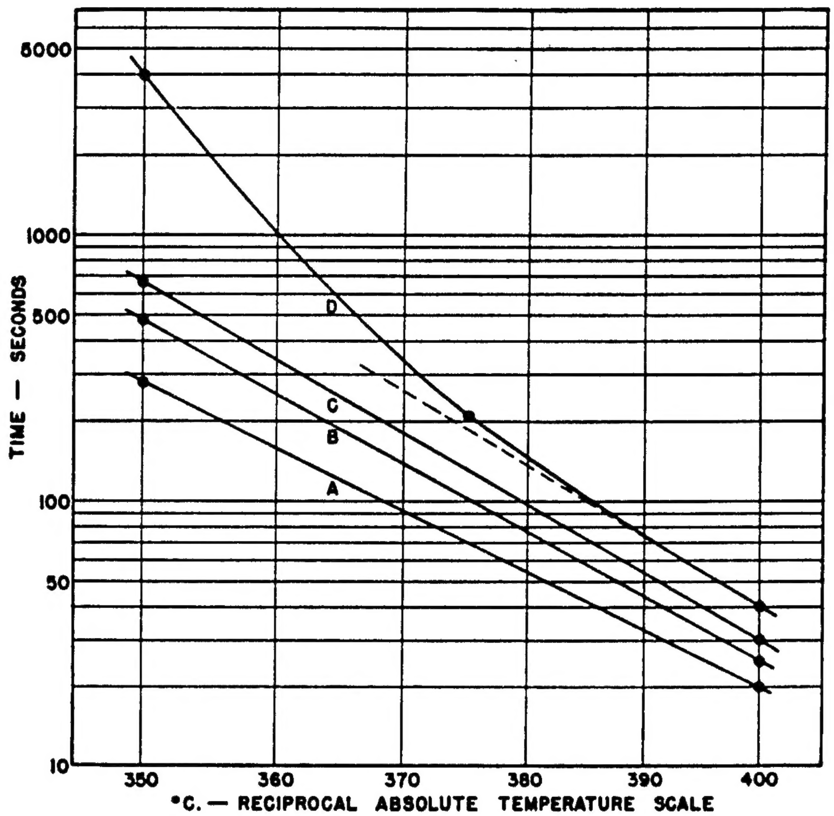


FIG 14—TIME FOR COMPLETE RECRYSTALLIZATION (LOG. PLOT) VS. TEMPERATURE (RECIPROCAL ABSOLUTE TEMPERATURE PLOT) FOR
A. High purity aluminum
B. Al + 0.025 pct Mg alloy
C. Al + 0.12 pct Mg alloy
D. Al + 2 pct Mg alloy

and 15, show that the effect, although small, definitely exists for the alloys investigated in the present work, even though the deformation used was as high as 33 pct by rolling.

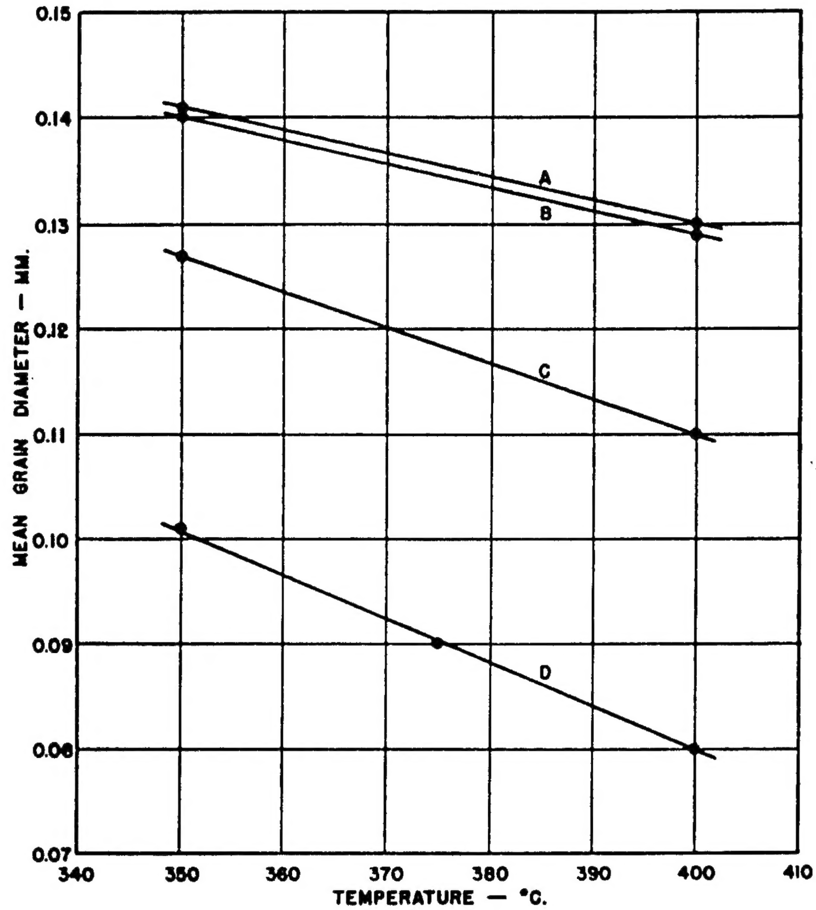


FIG 15—AVERAGE GRAIN DIAMETER, AS RECRYSTALLIZED, D_r, VS. TEMPERATURE OF RECRYSTALLIZATION FOR

A. High purity aluminum
B. Al + 0.025 pct Mg alloy
C. Al + 0.12 pct Mg alloy
D. Al + 2 pct Mg alloy

Conclusions

Isothermal grain growth data are presented for aluminum-magnesium alloys with 0.025, 0.12, and 1.8 to 2.05 pct magnesium, in the temperature range of 350 – 600°C from 20 sec to 11 days. The results indicate that the exponent n of the $D = kt^n$ relationship, which describes isothermal grain growth in these alloys, changes little up to 0.025 pct Mg, and then increases approximately linearly with the logarithm of the magnesium content, at least in the temperature range of 400 to 500°C. Thus, the first addition of 0.1 pct Mg in solid solution is relatively much more effective than further additions of the same amount.

The grain size, as recrystallized, at 350°C and 400°C, is also unaffected by the magnesium content up to 0.025 pct, and decreases approximately linearly with the logarithm of the magnesium content in the range of 0.025 to 2 pct Mg. The time for complete recrystallization at 400°C increases linearly with the logarithm of the magnesium content from 0.003 pct Mg (high purity aluminum) to 2 pct Mg.

At 350°C the time for recrystallization of the 2.05 pct Mg alloy is abnormally long, and the exponent n is abnormally low. It is probable that these effects result from the precipitation of a second phase.

Acknowledgments

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